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Microstructure Evolution at Ambient Temperature of 5N Purity Aluminum after Compression at Ambient and Liquid Nitrogen Temperatures

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In general, recrystallization occurs as a restoration process after deformation at a temperature ranging from (1/3)T_m to (1/2)T_m, where T_m is an absolute melting point. Occurrence of recrystallization depends on that of recovery as a competitive process. Aluminum is easy to recover after deformation and during deformation at elevated temperatures because of its high stacking-fault energy. In this study, microstructure evolution at ambient temperature after compression has been examined in 5N purity aluminum, for which an ambient temperature, 25°C (298K) is 0.32T_m. The cube specimen with all sides of 10mm was compressed by 50% at ambient and liquid nitrogen temperatures. As a result of analyses of microstructures before and after compression by SEM-EBSD technique, dynamic recrystallization during compression at ambient temperature was found. Static recrystallization was also observed after compression and holding for two hours. Further, relation between orientations before and after deformation or holding was analyzed.

Keywords: *ultra high purity aluminum, recrystallization, ambient temperature, EBSD analysis, compression.*

1. Introduction

In general, it is known that recrystallization temperature is (1/3)~(1/2)$T_{m,}$ where T_m is an absolute melting point [1, 2]. Especially, in higher purity metals static or dynamic restoration process can occur at a lower temperature. In a case of aluminum, (1/3)T_m is 38°C (311K), where its melting point is 660°C (933K). If ambient temperature is regarded as 25°C, restoration process or microstructural evolution in ultra high purity aluminum is expected with high possibility during holding or deformation at ambient temperature. Further, Schmidt and Haessner [3] reported that high purity aluminum deformed at 77K recovered and recrystallized below ambient temperature. It implies that deformation temperature greatly affects restoration temperature. In addition, aluminum is easy to recover during and after deformation at elevated temperatures because of its high stacking-fault energy. Then, interesting competition between recovery and recrystallization should be made in the restoration at ambient temperature.

In this study, 5N ultra high purity aluminum (99.999% Al) was compressed at ambient and liquid nitrogen temperatures to examine microstructure evolution at ambient temperature after compression by hardness test and SEM/EBSD analysis.

2. Experimental Procedure

A hot-rolled plate of 5N purity aluminum (99.999% Al) with a 12mm thickness was prepared. Cubic specimens with all edges of 10mm were made from the plate as each edge is respectively parallel to rolling direction (RD), transverse direction (TD) or normal direction (ND).

Specimens were compressed until a compressive strain of about 50% at ambient temperature (297K; Abbreviated as AT below) and liquid nitrogen temperature (77K; Abbreviated as LNT below). The Teflon sheet was inserted between the specimen and the upper and lower blocks for lubrication. To freeze microstructure after compression, the specimen was immersed into liquid nitrogen (77K) within 60 seconds. Then, the specimen was immersed for 180 seconds in ethanol at AT to reheat it to AT. After holding the specimen for a given time, microstructure evolution at AT after compression was examined by hardness test and SEM/EBSD analysis. Analyzed surface was electrochemical polished before compression.

Hardness test was conducted by micro-Vickers hardness tester at AT and a time ranging from 900s to 3.6Ms after compression. Test load was set as 2.94N (300gf) and hardness was calculated as the mean value of hardness numbers in 9 points sited as a lattice.

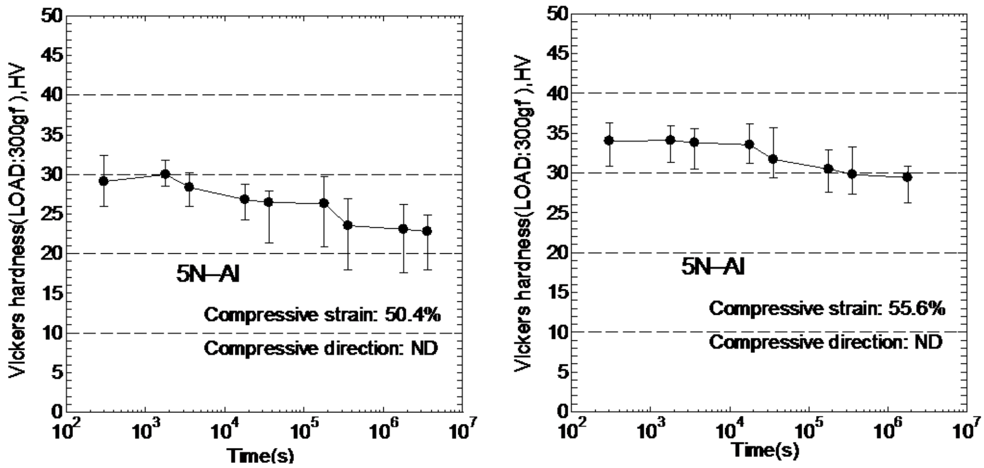
Scanning electron microscopy/ Electron back scatter diffraction (SEM/EBSD) was carried out on an area of 1000μm×1000μm at AT from 2 hours to1000 hours after compression.

3. Results and Discussion

3.1 Hardness during holding

Figure 1 shows change in micro-Vickers hardness during holding at AT after compression at ambient and liquid nitrogen temperatures. As shown in the figure, softening with increase of time happened at AT in 5N-Al compressed in normal direction (ND) to a compressive strain of 50.4%. This is probably because stored strain in compression was released by static recovery. The wide error bars of the hardness during softening were attributed to large difference in the stored energy depending on site in the sample, work hardening and static recovery. It is, therefore, confirmed by the hardness test that static restoration process occurs even at AT after compression at ambient temperature.

For the sample compressed at LNT, softening was also observed during holding at AT in 5N-Al compressed normal direction (ND) to a compressive strain of 55.6%. The hardness at 30s is 5HV higher than that for the sample compressed at AT because it was deformed at LNT. Although decrease in hardness showing static recovery proceeded at AT after LNT compression, the decrease was smaller than that for AT compression ones. This is because that strain release by dynamic recovery might not occur at LNT. But softening by static recovery process was the same as holding after AT compression, great stored energy have not be released.



(a) (b)

Fig. 1 Change in Vickers hardness at ambient temperature after compression at (a) ambient and (b) liquid nitrogen temperatures.

3.2 Load - Displacement Curve

Figure 2 displays load – displacement curves during compression at ambient and liquid nitrogen temperatures. The compressive load at LNT is about 10% higher than that at AT as shown in the figure. This is partially because the stored strain in compression was released by dynamic recovery or dynamic recrystallization during compression. It is, therefore, confirmed by the load– displacement curve that the sample compressed at LNT stored more strain than that for AT. The specimen with more strain is likely easy to recover or recrystallize at AT.

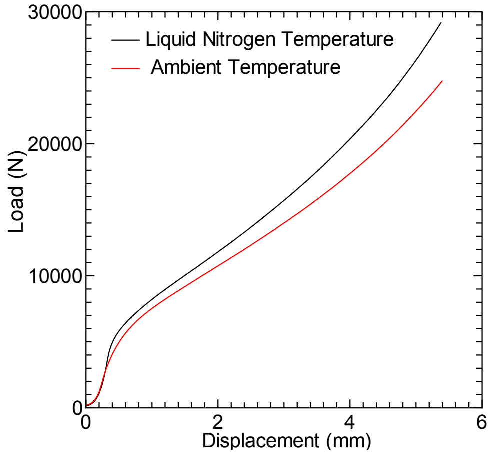
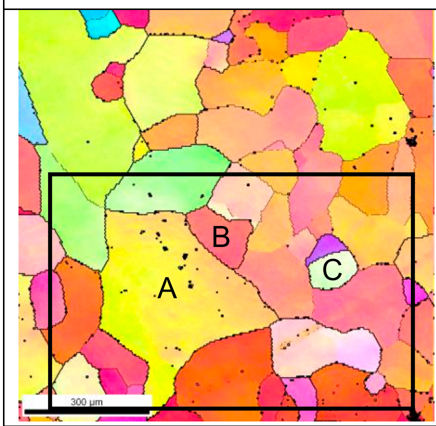
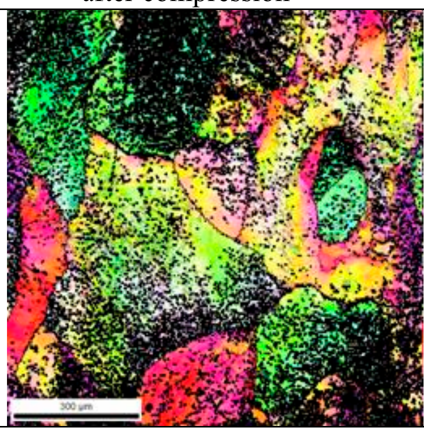


Fig. 2 Load–displacement curves during compression at ambient and liquid nitrogen temperatures.

3.3 Microstructure change at AT after AT compression

Obvious change in microstructure at ambient temperature is shown by inverse pole figure (IPF) maps in Fig. 2. The 5N-Al specimen was compressed in normal direction (ND) until a compressive strain of 55.4%. Before the compression, SEM/EBSD analysis on a certain area was performed, the area was repeatedly analyzed at given holding times at ambient temperature. A small square area in the map before compression corresponds to the analyzed area after compression. Comparison between the maps before and after compression revealed change in grain shape. As a result of examining changes in cross sectional areas of grains A, B and C, A became 1.96 times, B 2.13 times and C 1.93 times larger than before compression. Factors close to 2 almost corresponds to the compressive strain of 55.6%, which means an ideal compressive deformation with little friction. Then, after the compression local deformation is observed in the vicinity of C grain even though C grain itself deformed homogeneously (Fig. 2(b)). Further, microstructure change during restoration process is found at AT after AT compression. In Fig. 2(d) after 100 hours, new strain free grains, that is, recrystallized grains appear. Their grains grow at AT. This implies that static recrystallization happens at AT. Therefore, softening at AT after compression in Fig. 1 should be regarded as a result of static recrystallization.

(a) Before compression	(b) 1 hour(36000seconds) after compression
	
(c) 10 hours(36000seconds) after	(d) 100 hours(360000seconds) after

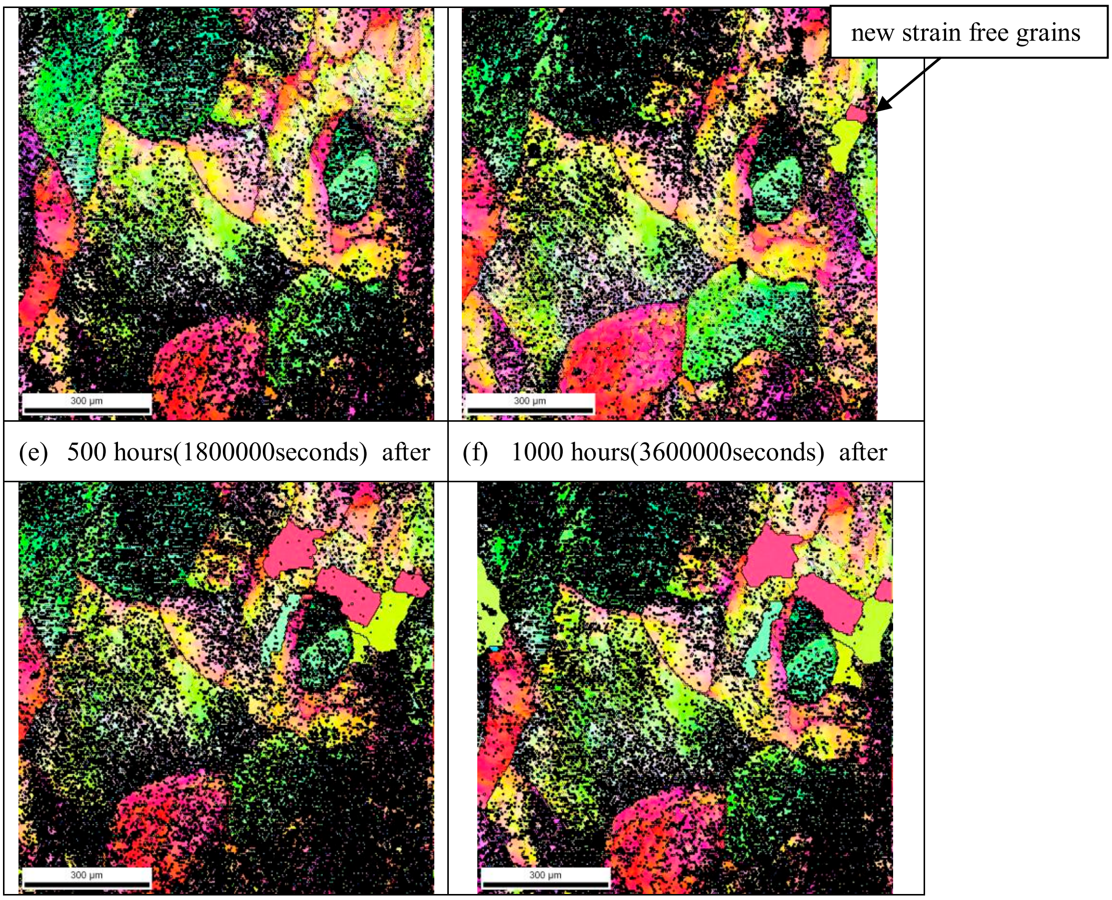


Fig. 3 Microstructure change at AT after AT compression.

3.4 Dynamic microstructural evolution of 5N-Al compressed at AT

Figure 3 exhibits microstructural dynamic evolution of 5N-Al compressed at AT. SEM/EBSD analysis examined in 5N-Al compressed normal direction (ND) until compression rate 53.1%. Many low angle boundaries are observed and some of them develop to high angle boundaries in black-circleded areas. In these areas, continuous dynamic recrystallization may take place.

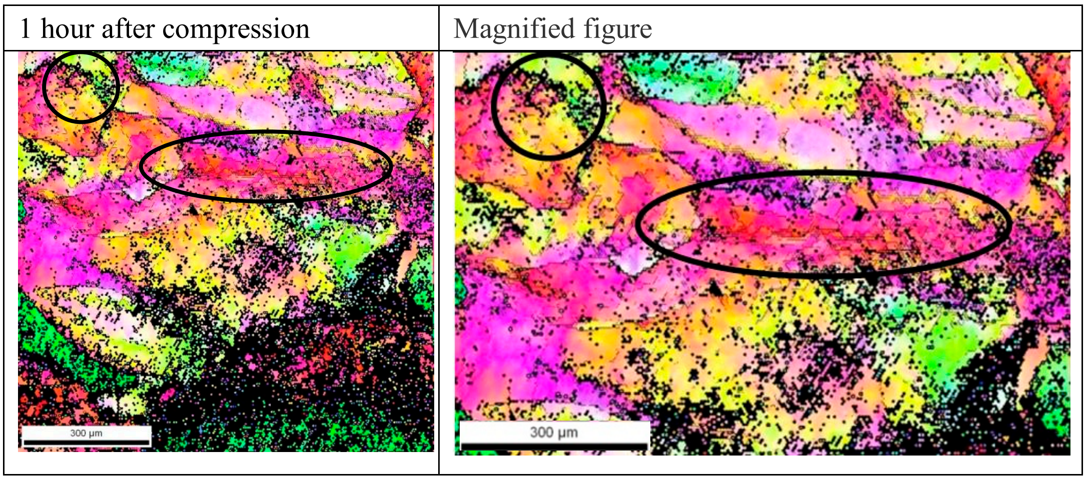


Fig. 4 Dynamic microstructural evolution of 5N-Al compressed at AT

4. Summary

In the present study, microstructure evolution at ambient temperature after compression has been investigated by hardness test and SEM/EBSD analysis. The results obtained are summarized as follows.

1. The hardness test revealed that softening with increase of time happened at AT in 5N-Al compressed in normal direction (ND) at AT and LNT. It is, therefore, confirmed that static restoration process occurs even at AT.
2. In the load – displacement curve, compressive load at AT was about 10% lower than that at LNT. It is, therefore, confirmed by the load– displacement curve that the sample compressed at LNT stored more strain than that for AT.
3. During holding at AT after compression at AT, new strain free grains appeared. Their grains grew at AT. This shows that static recrystallization takes place at AT.
4. In the dynamic microstructural evolution, many low angle boundaries were formed and some of them developed into high angle boundaries during compression at AT. In this areas, continuous dynamic recrystallization may occur.

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